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Quantitative studies of beam-induced defects in III–V compounds by cathodoluminescence and transmission electron microscopy

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Abstract

Electron beam currents above about 1 μA induce long straight dislocations lying just below the surface in (001) wafers of GaAs and InP. These dislocations are of interest in themselves and are well situated for cathodoluminescence (CL) dark contrast studies. Microcomputer Monte Carlo electron trajectory simulation-based programs were written to calculate emitted CL intensities and to simulate defect CL contrast profiles. Series of simulations for increasing beam energies (penetration range) show that the dislocation contrast is a maximum for a certain accelerating voltage which enables the dislocation depth to be determined. The magnitude of the contrast for a given beam energy increases with increasing defect recombination strength which can therefore be identified. The possibility of finding the number of dislocations in individual dark line bundles is discussed. The results of further transmission electron microscopy observations on the beam-induced dislocations are also presented.

1. Introduction

The cathodoluminescence contrast of dislocations has been relatively little studied compared with electron-beam-induced current (EBIC) contrast despite its importance, although that is beginning to change. There were, for example, five papers on combined EBIC and cathodoluminescence (CL) defect contrast work in the proceedings of the 1991 BIADS conference [1].

Franzosi *et al.* [2] found that beam currents above about 1 μA would induce dense grids mainly of edge-type dislocation lines parallel to the surface, running across the scanned area and with Burgers vector normal to the water surface in GaAs (001) and InP (001) material. This was ascribed to the production of high densities of vacancies of the volatile constituent under bombardment with subsequent aggregation to produce dislocations. These dislocations are unusual and interesting in themselves; their density can be controlled under the electron beam irradiation by on-line panchromatic CL imaging and are conveniently oriented for CL contrast studies. As an example, a panchromatic transmission cathodoluminescence (TCL) micrograph of electron-beam-induced dislocations in an Si-doped GaAs wafer is shown in Fig. 1. Dislocations aligned along the two ⟨110⟩ type directions can be seen.

A continuing analytical CL study of these beam-induced dislocations is being carried out and transmission electron microscopy (TEM) is being used to determine the nature of the defects and to study the way in which they are produced [3].

TEM observations show that the induced defects are single dislocations and give evidence of the bowing of dislocations where the energy dissipation volume

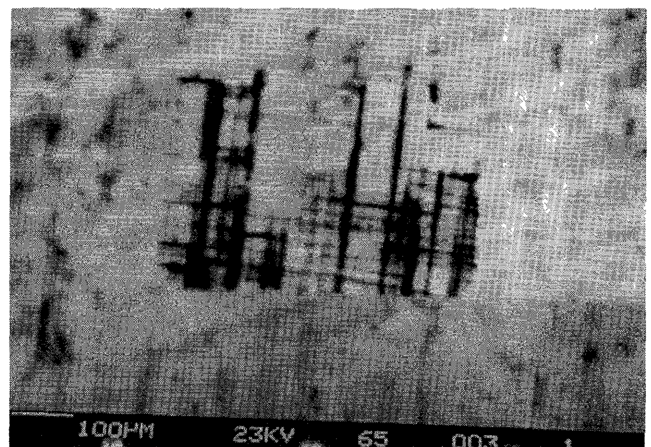

Fig. 1. TCL picture (Si detector) of electron-beam-induced dislocations in an Si-doped GaAs single crystal. The dislocations are aligned along the two ⟨110⟩ type conditions.

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under the beam has swept through them. This can be seen in the (001)-oriented transmission electron micrograph in Fig. 2. The picture also demonstrates that, owing to the difference between the scales of the emission cathodoluminescence (ECL) micrographs and the transmission electron micrograph, the CL contrast lines in Fig. 1 often correspond to groups of dislocations [3].

Further results of these studies are presented here. The mechanism of formation of beam-induced dislocations is discussed. It is found that there are large differences in the ease with which dislocations can be induced in different makes of scanning electron microscope and the reasons for this are discussed.

2. Experimental methods

A JEOL JSM-840A electron microscope fitted with a Matelect ISM-5 system to handle the signal from Si photodetectors was used for the ECL studies. A Cambridge Stereoscan 250 scanning electron microscope equipped with a panchromatic CL system (Si detector) produced by the EDS factory of Cuorgnè (TO) has also been used both for inducing dislocations and for controlling on line their propagation and density in the TCL mode. The TEM studies were carried out on a JEOL 2000FX electron microscope. The Monte Carlo programs run on IBM compatible microcomputers.

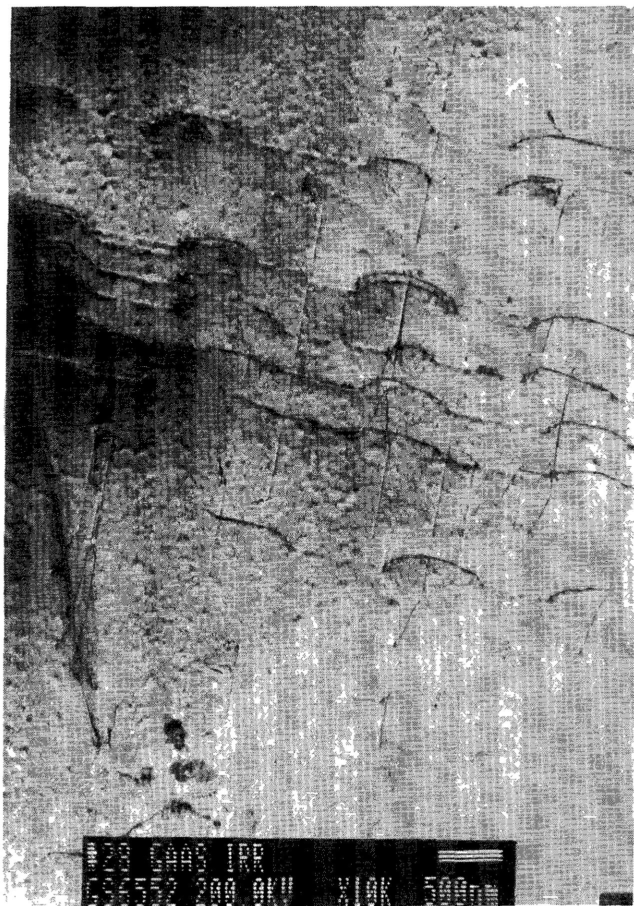

Fig. 2. (001)-oriented-plan view transmission electron micrograph of the same sample as in Fig. 1 ($\boldsymbol{g} = 131$).

3. Beam-inducing dislocations

It is found that dense grids of dislocations are readily induced by scanning with beam currents greater than about 1 μA in the Cambridge Stereoscan 250 at MASPEC whereas it is only with difficulty and at the highest attainable beam currents (above about 10 μA) that they can be induced in the JEOL JSM-840A instrument at Imperial College. It is thought that the current density may be a key parameter, and measurements of the beam diameters in the two instruments when operated in these high current conditions are now in hand to test this idea.

4. The CL programs

The Monte Carlo calculations use random numbers to determine the deflections of beam electrons penetrating into solid targets and so to calculate possible beam electron paths. Calculating sufficiently large numbers of these trajectories then yields statistically reliable distributions of the energy deposited in the material by the beam. The energy deposited at each point gives the rate of hole–electron pair generation, and multiplication by the radiative recombination probability gives the spatial distribution of the rate of emission of photons. Using a simplified model to take account of total internal reflection and Fresnel reflection at the exit surface and of absorption in the interior gives the emitted CL intensity L_{CL}. Thus the strength of the emitted CL as a function of the beam current and voltage and the materials or device parameters can be found. This was done for a number of bulk cases using this type of microcomputer program and the results of the simulations compared well with experimental data [4]. The results are also in agreement with earlier work using the results of Monte Carlo electron trajectory simulations and a more sophisticated calculation of CL intensities using mainframe computers [5]. A development of the microcomputer Monte Carlo program was then written to calculate dislocation CL contrast line scan profiles using the phenomenological model [6–8] originally developed for EBIC by Donolato [9]. The results obtained using this program were also compared with experimental results on

beam-induced dislocations [4]. Similar results have now been reported by the Singapore group [10] using mainframe programs.

By fitting experimental data to CL contrast profiles calculated in this way, a number of important quantities can be conveniently determined. For example the contrast will vary with beam voltage as the energy dissipation volume penetrates deeper into the material, moving the depth of greatest hole–electron pair density down relative to the dislocation depth (Fig. 3(a)).

Series of simulations for dislocations at the same depth but of different recombination strengths give families of curves. Experimental measurements of line scan widths (resolutions) and peak shapes of curves of C_{CL} *vs.* E_b (as in Fig. 3(a)) for different recombination strengths can be used to determine the latter parameter, even if care must be taken since more dislocations can pile up (Fig. 3(b)) in a cylinder of material smaller than the lateral CL resolution, so affecting the CL contrast.

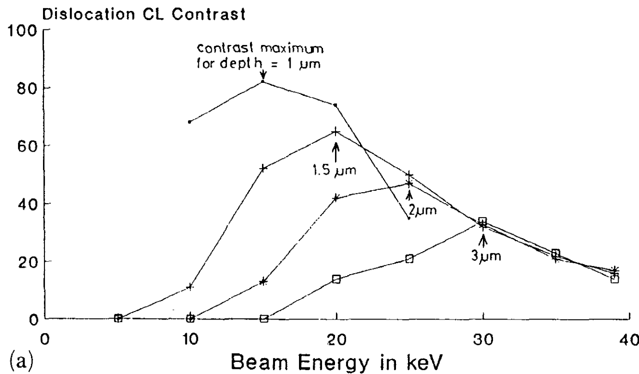

Fig. 3. (a) Dislocation CL contrast *vs.* beam energy for a dislocation cylinder of 1 μm radius in which 30% of hole–electron pairs recombine non-radiatively with dislocations lying at depths of 1, 1.5, 2 and 3 μm as marked. The CL contrast peak lies at a beam energy that increases with dislocation depth. By fitting experimental values to such families of curves the dislocation depth can be determined. (b) Bright-field (110) zone axis cross-section transmission electron micrograph of three beam-induced dislocations in an Si-doped GaAs single crystal.

5. Dislocation CL contrast

Figure 4(a) is a typical view of an area of a low density of beam-induced dislocations (the horizontal and vertical lines). The direction of scanning used in inducing the dislocations was at approximately 45° to this and a high dislocation density scanned area is visible at the top centre of the micrograph. For analysis, higher magnifications are used and well-resolved lines with contrast relatively uniform along their length are

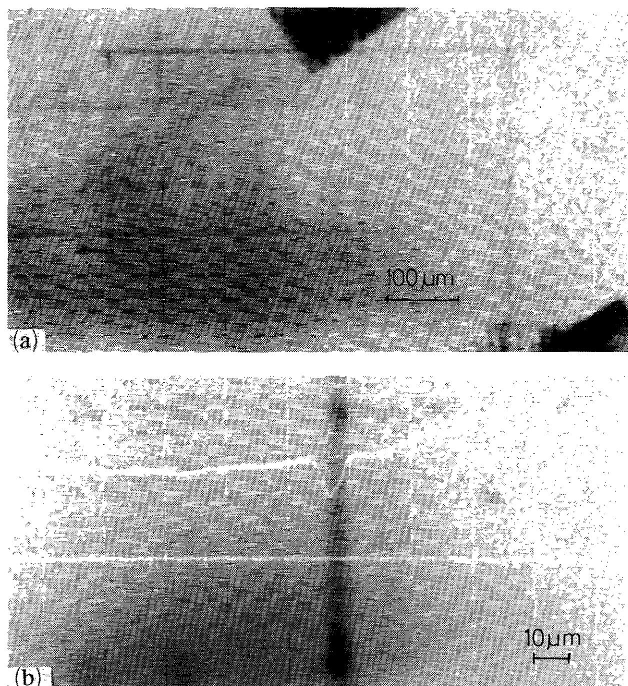

Fig. 4. (a) Low and (b) higher magnification ECL (panchromatic, essentially near band edge emission) micrographs of beam-induced dislocations in GaAs. (b) A CL intensity line scan profile is also shown that was recorded for a scan along the straight line recorded below it.

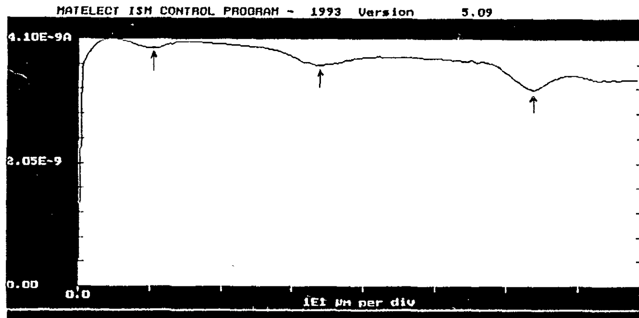

Fig. 5. Line scan profile of the photodiode (ECL) current for a line perpendicular to three parallel beam-induced dislocations (the three dips in the trace).

selected for the recording of line scan profiles as in Fig. 4(b). This illustrated a drawback of beam-induced defects as objects for analysis; some form of beam damage often results in varying background CL intensities so that the definition of contrast becomes difficult. Areas for analysis are selected to minimize this problem. Quantitative line scans can be recorded via the Matelect system as in Fig. 5. This showed that three parallel dislocations had similar values of contrast at 25 keV (3.6, 5.4 and 9.2%).

Two questions can now be addressed.

(i) Can the defect depth range be determined by measuring the CL contrast C_{CL}, as a function of beam voltage V_B, and is this depth constant?

(ii) Can the number of dislocations contributing to a particular dark line be determined by finding the recombination strength as $\gamma = n\gamma_D$ where γ_D is the recombination strength of a single dislocation and the minimum γ value found experimentally?

6. Discussion

Initial results of curve fitting suggest that dislocation depth can be determined to a few tenths of a micron. Determining the number of dislocations in a dark line bundle should also be possible. The three "dislocations" in Fig. 5 gave contrasts of $3.6 = 2 \times 1.8\%$, $5.4 = 3 \times 1.8\%$ and $9.2 = 5.1 \times 1.8\%$, suggesting that contrast does occur in discrete multiples of some single dislocation value. However, the gradual variation in brightness across the field of view by some 16% in Fig. 5, due to some form of beam damage, makes the accurate determination of dark line contrast values impossible. The phenomenon of damage will need to be investigated and brought under control to make quantitative CL contrast studies reliable.

Concerning the dislocation depth distribution, (110)-oriented cross-sectional TEM investigations provided evidence that dislocations were distributed

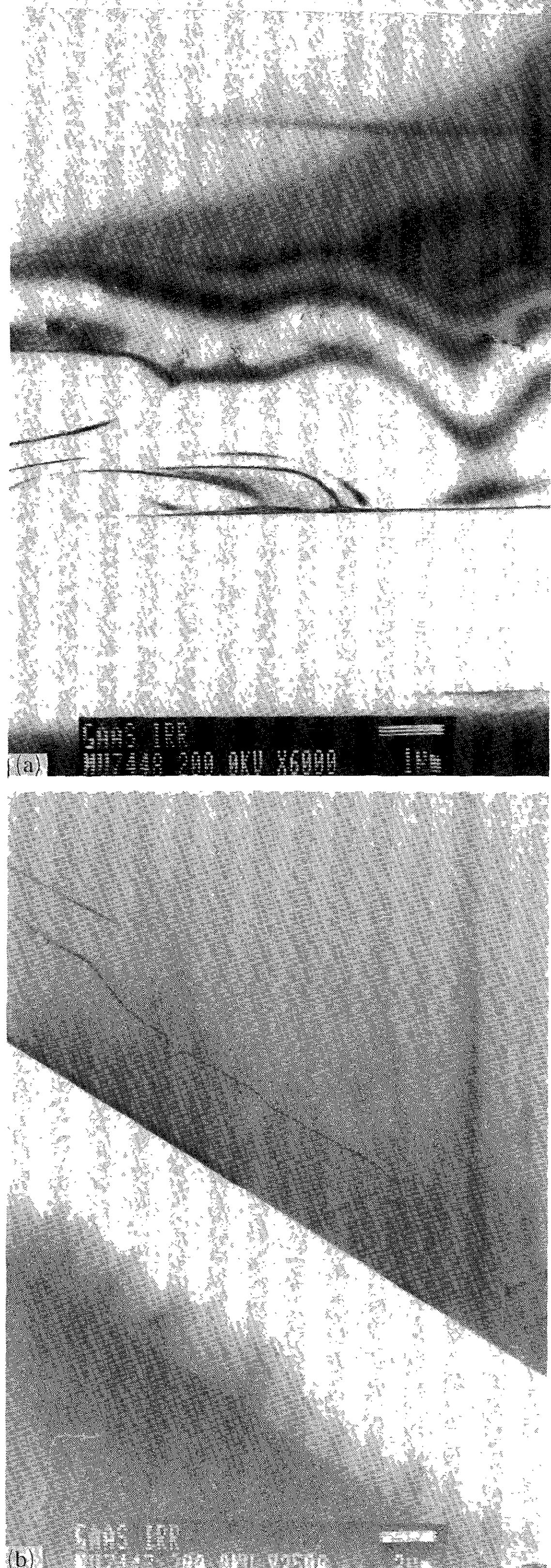

Fig. 6. Bright-field (110) zone axis cross-section transmission electron micrographs of GaAs wafers irradiated at (a) 10 keV and (b) 25 keV. Dislocations both almost perpendicular and parallel to the foil surface are shown at different depths.

from some tenths of a micron from the specimen surface to some microns inside the bulk wafer depending on the electron beam energy used during the irradiation. Figures 6(a) and 6(b) are transmission electron micrographs of different layers irradiated at 10 keV and 25 keV respectively. Dislocations emerging almost perpendicular to the foil surface and running parallel to the specimen surface are clearly shown at different depths; some dislocations propagating from the wafer surface are also shown.

In earlier work it was shown that, by fitting experimental results on defect-free material to curves calculated using these Monte Carlo programs, it is possible to determine several materials parameters rapidly. This approach can be applied to investigate the form of "damage", *e.g.* whether there is any change in the dead-layer thickness, or whether there is a change in form of the dependence of emitted CL intensity on beam energy for a constant beam current such as would arise from a change in radiative recombination probability (which relates the generated hole–electron pair density to the rate of photon emission from each depth) or a change in self-absorption coefficient.

Large changes in radiative recombination probability can arise from changes in the capture cross-sections of recombination centres. Electron bombardment and the consequent ionizing collisions can change the charge state of point defects and this can change their effective cross-sections by many orders of magnitude [11].

Although the phenomenological model of CL defect contrast is the same as that for EBIC [6–8] the results of CL contrast studies can be useful in several ways.

(i) Dark CL defect contrast can be used just like EBIC contrast to determine the recombination velocity (strength) and depth of the defect and to investigate its recombination properties.

(ii) The bright CL defect contrast which can occur at photon energies less than the band gap value presents the possibility of spectroscopy to determine defect energy levels.

It is intended to check the depth determined by CL contrast curves fitting against the depth found by cross-sectional TEM. The CL method of depth determination could then be useful for example for studies of strain relief in lattice-mismatched heterostructures.

While beam-inducing dislocations are attractive as a means for producing samples for CL contrast studies they also produce damage, but so, in many cases, does CL imaging.

Acknowledgments

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